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Lipid extraction and biodiesel production from municipal sewage sludges: A review

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ABSTRACT

Extensive research is being conducted all over the world to produce fuels from renewable biomass. Biodiesel, a renewable liquid fuel produced from lipid sources, is one of the most attractive among the options explored for alternative energy sources. However, 70–80% of the overall biodiesel production cost is associated with raw materials cost. Municipal sewage sludge is readily available at no cost. It contains various lipids and hence it is a promising raw material for biodiesel production. Lipids can be initially extracted from the sludge. Subsequently, the extracted lipid is converted to biodiesel by esterification and/or transesterification reaction. Biodiesel is also produced by in situ transesterification of dried sludge. This paper reviews the various lipid extraction techniques and biodiesel production processes from municipal wastewater sludge.

Contents

1. Introduction

Worldwide rapidly increasing fuel demand (85 million barrels of liquid fuel per day in 2006 and projected to increase to 107 million barrels of liquid fuel per day in 2030) and reducing fossil fuel reserve (1342 billion barrels oil as of January 2009) act as a driving force behind the search for alternative fuels [1]. The majority of all energy consumed worldwide is now coming from fossil fuel sources. Fossil fuel sources are non-renewable, and will be exhausted in near future [2]. Presently, there is an urgent need for alternative cheap and renewable energy resources with little or no environmental impact. The alternative fuel sources being developed around the globe include, such as biodiesel, alcohol, biomass, biogas, synthetic fuels. Among them biodiesel can be used directly, while others need some sort of modification before they are used as substitute of conventional fuels [3]. Biodiesel is renewable, biodegradable, less toxic, and safer for storage and handling, has excellent lubricity and could provide similar energy density to diesel [1,4,5]. It burns much cleaner than petroleum diesel as it contains oxygen and reduces most emissions (CO_2, CO, particulate, except NO_x) [6]. Biodiesel does not require new refuelling stations, new parts inventories or expensive engine modifications [7].

Chemically, biodiesel consists of fatty acid methyl esters that can be produced from various lipid sources by transesterification reaction with alcohol in the presence of a base, acid, enzyme or solid catalyst [1,5,8,9]. Depending upon the climate and soil conditions, different countries are looking for different types of vegetable oils as substitutes for diesel fuels. For example, soya bean oil in the US, rapeseed and sunflower oils in Europe, palm oil in south-east Asia (mainly Malaysia and Indonesia) and coconut oil in the Philippines are being considered [10]. A major economic challenge

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for the commercialization of biodiesel production is the high cost of pure vegetable or seed oils, which constitutes between 70% and 85% of the overall biodiesel production cost [1,11,12]. Moreover, the use of edible vegetable oils for biodiesel production has recently been of great concern. Used frying oil (UFO) for biodiesel production introduces other challenges due to the broad properties of UFO that may affect the biodiesel production consistency [1,9,13]. Biodiesel can also be produced from algae, which is produced naturally all over the world [10,14]. Besides, some species of plants yielding non-edible oils, e.g. jatropha, castor, neem, karanja and pongamia may play a significant role in providing alternative raw materials [1,15]. But cultivation of these alternative raw materials requires huge land. Thus, in order to reduce the cost per gallon of biodiesel, alternative feedstocks that are readily available in large quantities and at low cost must be considered.

Sewage sludge is an abundant organic waste or by-product generated in wastewater treatment plant (WWTP) facilities after primary and secondary treatment processes [16–18]. A wastewater treatment facility having an activated sludge process produces two main types of sludge- primary sludge, a combination of floating grease and solids, and secondary sludge, composed mainly of microbial cells and suspended solids produced during the aerobic biological treatment. The primary sludge is collected at the bottom of the primary clarifier and the secondary sludge, known also as activated sludge, is collected in the secondary clarifier [12,17]. The management (handling, treatment, and disposal) of sludge is a complex challenge for any WWTP and contributes to 20–60% of total operating costs of WWTP [19].

Municipal wastewater treatment plants produce huge amounts of sludge per year that is readily available at free of cost or even with an incentive. Wastewater treatment plant facilities in USA alone produced over 6.2 million metric tons of dry sludge every year [20]. Six wastewater treatment plants in London, ON, Canada produced 3.8×10^5 m^3 wastewater sludge in 2008 [21]. The amount of sewage sludge is expected to increase in the future due to increasing urbanization and industrialization. The use of sludge as a fertilizer is restricted in many countries of world due to bad odour, the presence of heavy metals, toxic substances and pharmaceutical chemicals, while the sludge incineration results in emissions that contain heavy metals and dioxins [22,23]. The disposal of sludge in landfills involves with inherent chemical energy loss and associated health problems. The ocean dumping of sludge locally interrupts the ecology of biosphere [24]. One viable alternative to sludge management and disposal challenge is to utilize the sludge as a source of lipid feedstock for biodiesel production.

Lipid is a natural mixture of triglycerides, diglycerides, monoglycerides, cholesterols, free fatty acids, phospholipids, sphingo-lipids, etc. [25]. The municipal wastewater sludge contains a significant amount of lipid fraction that is a composite organic matrix (characterized as oils, greases, fats and long chain fatty acid) originating from the direct adsorption of lipids from domestic and industrial wastes in the sludge, and/or from the phospholipids in the cell membranes of microorganisms, their metabolites and by-products of cell lysis.

Research has indicated that the lipids contained in sewage sludge are a potential feedstock for biodiesel [18,20]. The overall biodiesel production scheme can be shown in Fig. 1. To avoid the interference in the biodiesel synthesis, lipids are usually extracted from the sludge with organic solvents. Several options have been attempted. However, the lipid extraction for biodiesel production from municipal sewage sludge poses great challenges for commercial realization. The main challenges include (1) the pre-treatment of raw sludge for efficient lipid extraction, (2) the lipid extraction from the sludge, (3) the biodiesel production methods from solid sludge, (4) the quality of biodiesel, and (5) process economics and safety.

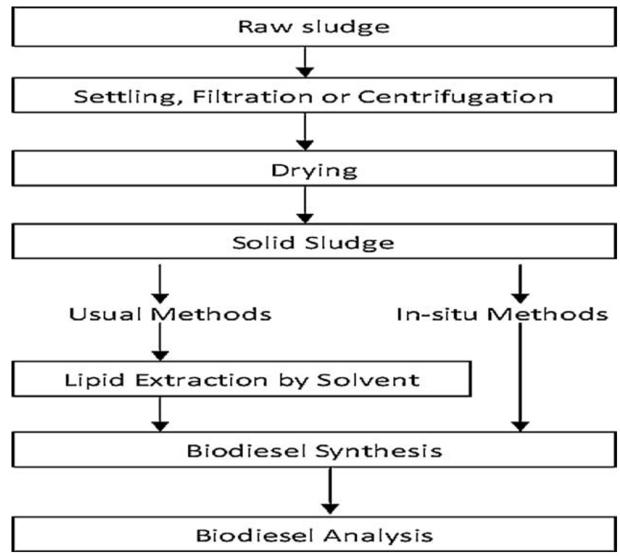

Fig. 1. Overall biodiesel production scheme.

2. The pre-treatment of raw sludge for efficient lipid extraction

Raw primary sludge and secondary sludge contain 4–5% (w/v) and 1–2% (w/v) solids, respectively. The lipid extraction from the raw sludge requires huge amount of organic solvent and large vessels with stirring and heating system. Dewatered concentrate sludge is sticky and hinders the lipid extraction process. But the lipid extraction from dried sludge is feasible.

Dufreche et al. [20] pre-treated the secondary sludge (2% solid) by gravity settling followed by centrifugation or pressure filtration. They performed centrifugation at 3000 rpm for 20 min and got 7–8% (w/w) solids containing sludge. They also conducted pressure filtration by using 80 μm and 20 μm nylon filter and got 12-14% solids. They used Hydromatrix to absorb residual free water. But this technique is challenging for large-scale pre-treatment of raw sludge. Mondala et al. [12] concentrated the raw primary and secondary sludge by gravity settling at 0 °C for 24 h. They further dewatered the sludge by centrifugation at 3000 rpm for 20 min and dried the sludge by freeze drying. Revellame et al. [1] recently used this technique for secondary sludge only. But this pre-treatment process is not feasible for large-scale production. Boocock et al. [26] extracted lipids from dried sludge supplied by Wastewater Technology Centre of Environment Canada. Recently Pokoo-Aikins et al. [17] used the sewage sludge for lipid extraction and biodiesel production, but they did not mention the type of sludge they used. The pre-treatment of raw sludge can significantly affect the lipid extraction process and consequently the yield of biodiesel production.

3. The lipid extraction and lipid analysis methods

Lipid extraction is the first step for biodiesel production from wastewater treatment plant sludge. At present, several methods are available for lipid extraction from biological materials. Most of these methods use organic solvents, usually in mixtures, as in the Bligh and Dyer [27] and Folch et al. [28]. Boocock et al. [26] extracted 12 w% lipids by soxhlet extraction method and 17–18 w% lipids by boiling solvent extraction from raw sewage sludge. They used a sludge to solvent ratio of 1 to 6 with 300 ml solvent (chloroform or toluene) for 50 g dry sludge and 600 ml solvent (chloroform or toluene) for 100 g dry sludge in soxhlet extraction method and boiling extraction method, respectively. They concluded that both chloroform and toluene are equally effective for lipid extraction,

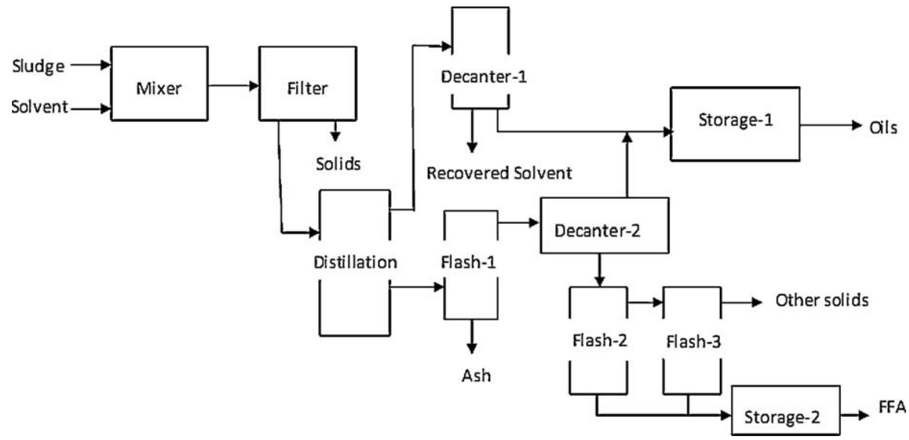

Fig. 2. Sewage sludge extraction process with hexane or toluene as solvent Pokoo-Aikins et al. [17].

but preferred toluene based on cost and environmental considerations. Approximately 65 w% of the extracts were found to be free fatty acids (C12-C18, mostly palmatic, stearic and oleic acid), 7 w% were glyceride fatty acids and 28 w% unsaponifiable material (C9 to C16 alkane).

Dufreche et al. [20] extracted lipids from activated sludge by using pure and/or mixture of hexane, methanol and acetone as solvents and obtained a maximum 27.43 ± 0.98% lipid for three times extraction by using 60 v% hexane/20 v% methanol/20 v% acetone. They also used supercritical-CO_2 technique and supercritical-CO_2 with methanol co-solvent technique for lipid extraction and obtained 3.55 w% and 13.56 w% lipid, respectively. They claimed that extraction of lipids by a mixture of n-hexane, methanol and acetone gave the largest conversion to biodiesel compared with other solvent systems.

Pokoo-Aikins et al. [17] extracted lipids from sewage sludge by using toluene, hexane, ethanol and methanol. They used a sludge to solvent ratio 1:5 that was calculated according to Boocock et al. [26]. They separated the free fatty acids (FFA), triglycerides (oils) and the solvent (Fig. 2) and found the yield of FFA was 24.8 w%, 24.9 w%, 25.5 w%, 25.5 w% for toluene, hexane, methanol and ethanol, respectively. The maximum yield of triglyceride was 3.4 w% for all four solvents.

4. The biodiesel production methods from solid sludge

There are four primary techniques for biodiesel production – direct use and blending of raw oils, micro-emulsions, thermal cracking and transesterification [9]. The most commonly used method for biodiesel production is transterification (also known as alcoholysis) reaction in presence of a catalyst. Transesterification is the process of exchanging the alkoxy group of an ester compound with another alcohol (Fig. 3).

The R_1, R_2, and R_3 are long hydrocarbon chains, called fatty acid chains [29]. This transesterification reaction consists of a series of consecutive, reversible reactions [30–32]. The triglyceride is converted stepwise by reacting with primary alcohol to diglyceride, monoglyceride and finally glycerol.

$$\text{Triglyceride} + \text{ROH} \leftrightarrow \text{Diglyceride} + \text{RCOOR}_1$$

$$\text{Diglyceride} + \text{ROH} \leftrightarrow \text{Monoglyceride} + \text{RCOOR}_2$$

$$\text{Monoglyceride} + \text{ROH} \leftrightarrow \text{Glycerine} + \text{RCOOR}_3$$

The primary alcohol used to form the ester is a major feedstock in biodiesel production process. Methanol is widely used as the alcohol for producing biodiesel because it is the least expensive alcohol. Besides it has some advantages – (i) its reactivity is high, (ii) it does not absorb water that interferes with transesterification reaction, (iii) prevents soap formation and (iv) its recovery is easier, as it does not form azeotrope like ethanol [33,34]. Excess methanol (60–100% more methanol than required) is added in order to ensure total conversion of the vegetable oil or animal fat to its esters.

The yield of transterification depends on several factors including the type of catalyst (base, acid, enzyme or heterogeneous), alcohol/vegetable oil molar ratio, temperature, and duration of reaction, water content and free fatty acid content. Water can consume the catalyst and reduce catalyst efficiency.

Base catalyst transesterification is widely used commercially due to very fast reaction rate compared to other catalysts. Base

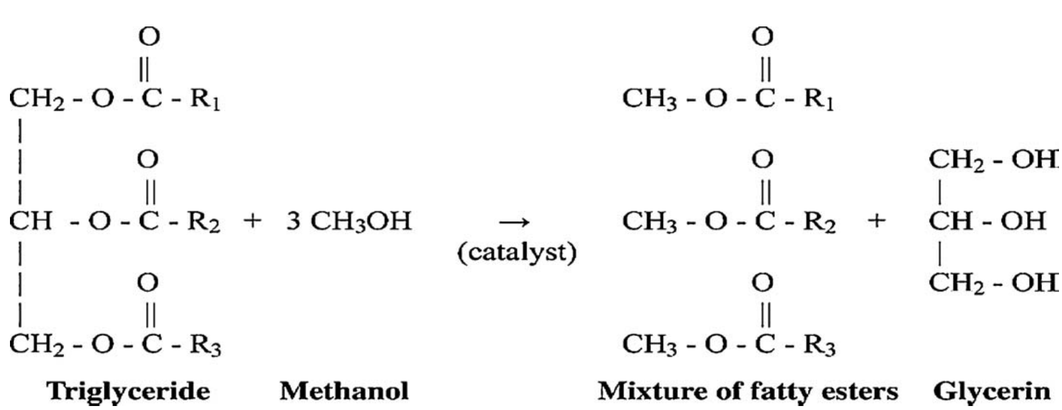

Fig. 3. The general form of transesterification reaction.

Table 1
Comparison of different technologies for biodiesel production [42].

Variable	Base catalyst	Acid catalyst	Lipase catalyst	Supercritical alcohol	Heterogeneous catalyst
Reaction temperature (°C)	60–70	55–80	30–40	239–385	180–220
Free fatty acid in raw materials	Saponified products	Esters	Methyl esters	Esters	Not sensitive
Water in raw materials	Interfere with reaction	Interfere with reaction	No influence		Not sensitive
Yield of methyl esters	Normal	Normal	High	Good	Normal
Recovery of glycerol	Difficult	Difficult	Easy		Easy
Purification of methyl esters	Repeated washing	Repeated washing	None		Easy
Production cost of catalyst	Low	Low	Relatively high	Medium	Potentially low

catalyzed process is very sensitive to water and free fatty acids (FFA) present in lipid sources due to soap formation. It leads to catalyst consumption and soap formation that inhibits separation of the glycerol from the methyl esters and contributes to emulsion formation during the water wash.

$$\text{Fatty acid}\,(R_1COOH) + NaOH \leftrightarrow \text{Soap}\,(R_1COONa) + \text{Water}\,(H_2O)$$

On the other hand, acid catalyzed transterification is 4000 times slower than base catalyzed transesterification and requires high triglyceride to alcohol ratio [35]. But acid catalyst is able to catalyze both the esterification and transesterification and more biodiesel is produced. If water accumulates, it can stop the reaction. So, this approach requires a water management technique.

$$\text{Fatty acid}\,(R_1COOH) + \text{Alcohol}\,(ROH)$$
$$\leftrightarrow \text{Mono-alkyl-ester}\,(R_1COOR) + \text{Water}\,(H_2O)$$

The use of heterogeneous catalysts has shown greater promise toward transesterification to obtain biodiesel. The commonly used heterogeneous catalysts are Mg/La mixed oxide, S-ZrO_2 sulfated zirconia, KOH/Nax zeolite, Li/CaO, CaO, KI/Al_2O_3, (ZS/Si) zinc stearate immobilized on silicagel, KNO_3/Al_2O_3, $SO_4{}^{2-}/TiO_2–SiO_2$, etc. Heterogeneous catalysts can be recovered conveniently from reaction products [9,36]. The undesired saponification reactions can be avoided by using heterogeneous acid catalysts. This catalyst enables the transesterification of vegetable oils or animal fats with high contents of free fatty acid (FFA) [37]. Solid catalyst can be reused and provides the possibility for carrying out both transesterification and esterification reactions simultaneously [38]. However, the cost of solid catalyst is very high.

Enzymatic transesterification looks attractive and encouraging for reasons of ease of product separation, minimal wastewater treatment needs, easy glycerol recovery and the absence of side reactions [39]. However, enzymatic process has several technical difficulties such as slow reaction rate, contamination of the product with residual enzymatic activity and high cost [10].

Kusdiana and Saka [31] developed a supercritical methanol technique in which biodiesel and glycerol were produced by the trans-esterification reaction of the raw oils and fats in absence of a catalyst. Biodiesel was also generated simultaneously by the esterification reaction of the free fatty acids, even if there was a high content of free fatty acids in the raw material oils and fats. Biodiesel was derived at high yield with no saponified products. Moreover, the separation and purification were easy because of the non-catalytic process. However, this method required extreme temperature and pressure conditions of 350 °C and 43 MPa, respectively, and induced breakdown of unsaturated fatty acids and trans isomerization, leading to adverse effects on the fluidity of the fuel at low temperatures [40,41]. The comparison of different technologies for biodiesel production is shown in Table 1.

Dufreche et al. [20] produced 4.41% biodiesel (based on total dry weight of activated sludge) from the solvent extracted lipids by acid catalyzed esterification-transesterification reaction. They also produced 6.23 w% biodiesel from the dried secondary sludge by in situ acid catalyzed transesterification. Mondala et al. [12] investigated the potential of primary and secondary sludge of municipal wastewater treatment plant as a lipid feedstock for biodiesel production by acid-catalyzed in situ transesterification process. They obtained at maximum FAME yield of 14.5 w% and 2.5 w% for primary and secondary sludge, respectively at 75 °C, 5% (v/v) H_2SO_4, and 12:1 methanol to sludge mass ratio.

Revellame et al. [1] optimized the in situ transesterification of activated sludge by full factorial design of four temperature levels (45, 55, 65, and 75 °C), six methanol to sludge ratio (5, 10, 15, 20, 25, and 30, v/w) and five levels of catalyst concentration (0.5, 1, 2, 4, and 6 v%). They obtained 4.88 w% and 4.79 ± 0.02 w% biodiesel, respectively, by numerical and experimental optimization at 55 °C, 25 methanols to sludge ratio and 4 v% sulphuric acid. They concluded that biodiesel production decreases significantly at temperatures above 60 °C due to acid catalyzed polymerization of unsaturated fatty acids or their ester.

5. The quality of biodiesel

The biodiesel must satisfy the ASTM D 6751 Standard Specification for Biodiesel Fuel (Table 2) in order to be used in an engine without problems. Ensuring the quality of wastewater sludge biodiesel is a great challenge. In addition to triglyceride, fatty acids, phospholipids, bacterial lipids, sludge may also contain various chemicals like wax esters, steroids, terpenoids, polyhydroxyalkanoates, hydrocarbons, linear alkyl benzene, polycyclic aromatic hydrocarbons, pharmaceutical chemicals, etc. [1,18,23,43,44]. These compounds may be extracted during the lipid extraction or in situ biodiesel production and contribute to the overall gravimetric yield. Conversion of these contaminants to biodiesel via cracking process will significantly increase fuel yield from the wastewater sludge [1,45,46].

Dufreche et al. [20], Mondala et al. [12], and Revellame et al. [1] reported that biodiesel from wastewater treatment plant sludge mainly contains methyl esters of palmatic acid (C16:0), palmitoleic

Table 2
ASTM D 6751 requirements [42].

Property	Method	Limits	Units
Flash point, closed cup	D 93	130 min	°C
Water and sediment	D 2709	0.050 max	v%
Kinematic viscosity, 40 °C	D 445	1.9–6.0	mm^2/s
Sulfated ash	D 874	0.020 max	w%
Total sulfur	D 5453	0.05 max	w%
Copper corrosion strip	D 130	No. 3 max	
Cetane number	D 613	47 min	
Cloud point	D 2500	Report to customer	°C
Carbon residue	D 4530	0.050 max	w%
Acid number	D 664	0.80 max	mgKOH/g
Free glycerine	D 6584	0.020	w%
Total glycerine	D 6584	0.240	w%
Phosphorous	D 4951	0.0010	w%
Vacuum distillation end point	D 1160	360 °C max, at 90% distilled	°C
Storage stability	To be determined	To be determined	To be determined

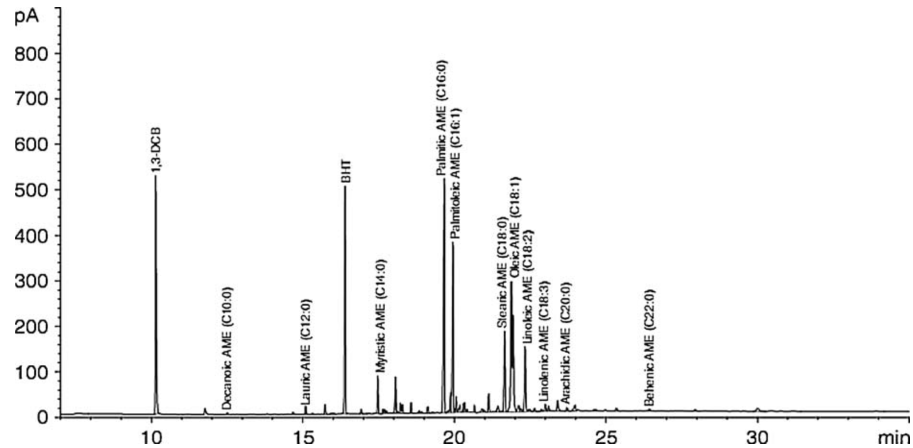

Fig. 4. FAME analysis of biodiesel obtained produced by in situ transesterification of secondary sludge [1].

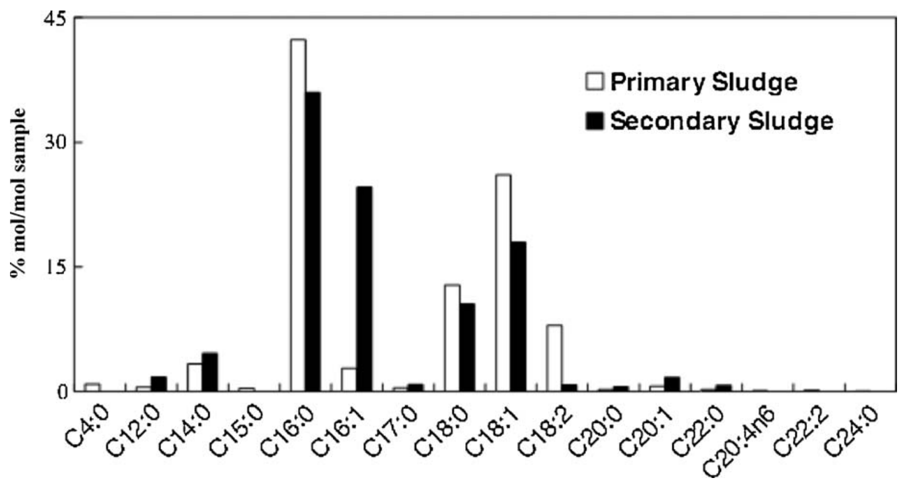

Fig. 5. FAME analysis of biodiesel obtained from in situ transesterification of municipal primary and secondary sludge at 4 h reaction time, 50 °C, 5% (v/v) H_2SO_4, and 8:1 methanol to sludge mass ratio [12].

acid (C16:1), stearic acid (C18:0), oleic acid (C18:1) and linoleic acid (C 18: 2) that are similar to pure vegetable oil biodiesel composition (Fig. 4).

Mondala et al. [12] also found the close similarity of the fatty acid composition of primary and secondary sludge by GC analysis (Fig. 5). Therefore it might be feasible to combine the two sludge types into a single feedstock for biodiesel production. But lipid percentage of the mixed sludge will be changed and process parameters need to be changed accordingly.

6. Process economy

Biodiesel is attractive due to environmental benefits and the fact that it is produced from renewable resources. However, the challenges are its cost and limited availability of fat and oil resources. The biodiesel production cost consists of two major cost items: the cost of raw material (fats and oils) and the operating costs. The cost of raw materials accounts for 70–80% of the total cost of biodiesel fuel [1,47]. Higher production cost has hindered the biodiesel growth and made it uncompetitive compared to diesel.

The biodiesel production from municipal sewage sludge can lower the cost significantly. Lipid extraction and biodiesel production from sewage sludge is associated with the use of organic solvents. But more than 99% of the solvents are recoverable [17]. The estimated biodiesel production cost from municipal primary and secondary sludge is USD 3.11 to USD 3.23 per gallon of biodiesel compared to USD 4.00 to USD 4.50 per gallon refined soy biodiesel and USD 3.00 per gallon for diesel (as of January 2010) [12,17,20,48]. Pokoo-Aikins et al. [17] claimed that the overall biodiesel production cost from free sewage sludge is varied according to the solvent used in initial extraction step and is USD 3.39 per gallon for ethanol, USD 3.37 per gallon for methanol, USD 2.89 for hexane, and USD 2.79 per gallon for toluene used as the extraction solvent. Although toluene is cheaper, recovery of it is more energy intensive due to its higher boiling point. Mahamuni and Adewuyi [49] reported that the use of high-frequency ultrasound significantly reduces the biodiesel production cost.

Table 3
Production cost estimate for sludge biodiesel [20].

	Cost per gallon (US $)
Centrifuge O&M	0.43
Drying O&M	1.29
Extraction O&M	0.34
Biodiesel processing O&M	0.60
Labor	0.10
Insurance	0.03
Tax	0.02
Depreciation	0.12
Capital P&I service	0.18
Total cost	3.11

Assuming 7.0% overall transesterification yield. O&M: operation and maintenance; P&I: protection and indemnity.

Dufreche et al. [20] assumed 7% overall biodiesel yield and estimated the production cost for sludge biodiesel (Table 3). Biodiesel production cost from sludge (USD 3.11 per gallon) is broken down to USD 2.06 per gallon for centrifuge, drying, and extraction processes and to USD 1.05 per gallon for other expenses. But they did not include the cost recovered by glycerol sale that will reduce the biodiesel production cost. After lipid extraction the valuable chemicals recovery from the sludge will be easier which may reduce the biodiesel production cost as well.

7. Summary

Biodiesel occupies a prominent position as a renewable liquid fuel. It has several benefits over diesel fuel. But higher production cost due to raw materials has made it uncompetitive compared to petro-diesel. Moreover, vegetable oils and animal fats which are the main raw materials for biodiesel production compete with food materials causing the cost of biodiesel to increase. Municipal sewage sludge is readily available and is a potential source of lipid for biodiesel production. But there are few challenges for biodiesel production from sludge.

First, the pre-treatment of raw sludge which includes collecting, dewatering and drying of sludge is quite costly. Freeze drying system to get dry sludge is energy and time consuming. Vacuum drying or other techniques can be used instead of freeze drying which will reduce the overall biodiesel production cost. Second, lipid extraction from sludge is also expensive and requires large volume of organic solvents. The amount of lipid depends on the sources and type of sludge. Solvent selection, sludge to solvent ratio, extraction time, temperature and solvent recovery are among the factors that affect lipid extraction efficiency and cost. Optimization of these factors is necessary for efficient lipid extraction. Lipid extraction by super critical CO_2 is not efficient due to higher operating costs. Microwave lipid extraction technique can be used which is rapid, safe, cheap and does not require samples devoid of water [50]. Third, biodiesel production methods are varied and often costly. The catalyst requirement for acid catalyzed in situ transesterification is high. Acid catalyzed transesterification is slow. Moreover, water produced during esterification of FFA makes it even slower, and hence acid catalyzed esterification followed by base catalyze transesterification can be used for biodiesel production from sewage sludge. But optimization of the amount of alcohol, catalyst (both acid and base), catalyst neutralization, biodiesel washing and drying is necessary for acid catalyzed esterification followed by base catalyzed transesterification. Heterogeneous catalysts, enzymatic catalysts or non-catalytic supercritical methods which are not affected by FFA or water can also be used for biodiesel production. Extensive research work is necessary to get optimum method for biodiesel production from municipal sewage sludge and to make it profitable compared to other sources.

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